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Longitudinally Detected ESR Magnetometer for Wide-Range Measurements of Low Fields

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A new magnetometer utilizing a longitudinally detected ESR (LODESR) method was developed. The probe head of the LODESR magnetometer is equipped with a single-turn coil (8 mm in diameter) which has a very wide bandwidth because the reactance of the coil is always smaller than the resistance of the transmission line (50 ohm) at frequencies less than 700 MHz. Thus, an absolute magnetic field could be measured over a wide range (2 to 9 mT) using this magnetometer without changing the probe head. © 2001 Academic Press

Key Words: **longitudinally detected ESR; magnetometer; magnetic field measurements; wide-range measurements; single-turn coil.**

INTRODUCTION

Absolute magnetic field measurements in low fields less than 10 mT are important for calibrating electromagnets, such as the field gradient coils of magnetic resonance imaging systems. For this purpose, an ESR magnetometer has been employed (*1–3*). A method that uses this magnetometer is based on measuring the resonant frequency, f_0, of the electron spins, which is related to the static magnetic field, B_0, by $B_0 = hf_0/g\beta$ (h, Planck's constant; β, Bohr magneton). By using this equation, the absolute magnetic field can be calculated from the resonant frequency because the g-value is barely affected by variations in some parameters, such as temperature. For measuring wide range B_0, the resonant frequency varies widely; therefore, the magnetometer's probe head should have a sufficiently wide bandwidth. By utilizing the ESR magnetometer, the resonant frequency is determined from the frequency at which the radio waves are absorbed; so it is necessary to use the probe head where this absorption can be detected. When a resonator is used for this purpose to obtain sufficient sensitivity, the probe head must have a very narrow bandwidth. To overcome this problem in the resonator, an untuned probe head with a delay-line structure was designed (*2, 4*). Despite employing the delay line, the plural probe heads must be changed to measure fields less than 10 mT (*2*). Thus, it is difficult to measure a wide-range B_0 with the ESR magnetometer.

The longitudinally detected ESR (LODESR) method is one of the techniques designed to observe ESR. With this technique, the signal is derived from a longitudinal (i.e., parallel to B_0) oscillation of the magnetization caused by spin flipping under on/off modulated ESR irradiation (*5–9*). Because the radio waves are only necessary to excite the electron spin of the paramagnetic species in LODESR, a device such as the resonator is not always necessary. A single-turn coil can be used to irradiate the electron spins with radio waves. Because the current in a single-turn coil that has a relatively small diameter approximates to the short current in a transmission line over a wide RF range, the bandwidth of the coil is very wide (*9*). In this study, a new magnetometer utilizing the LODESR method was developed. Because a probe head equipped with a single-turn coil has a wide bandwidth, it was possible to measure a wide range of B_0.

RESULTS AND DISCUSSION

LODESR Magnetometer

The block diagram of our LODESR magnetometer is shown in Fig. 1. Its probe head consists of a paramagnetic species, an irradiation coil, and a pickup coil.

The paramagnetic species is irradiated with radio waves by using the irradiation coil. When the radio waves are on/off modulated under ESR conditions, the longitudinal oscillation of the magnetization of the paramagnetic species is detected by the pickup coil. The frequencies of the radio waves are swept, and then one searches for the peak of the LODESR signal of the paramagnetic species. The frequency at which this peak appears is the resonant frequency of the paramagnetic species. The absolute B_0 strength is obtained from the resonant frequency and the g-value of the paramagnetic species by using the abovementioned equation.

Fifty milligrams of 1,1-diphenyl-2-picrylhydrazyl (DPPH) was used as the paramagnetic species. Its g-value is 2.0036 (*10*). The anisotropy of the g-value of the DPPH powder was not observed at resonant frequencies less than 700 MHz.

The irradiation coil is a single-turn coil with a diameter of 8 mm. It is made of copper wire (1 mm in diameter) and soldered to a semirigid coaxial cable (3.5 mm in diameter), which has a characteristic impedance of 50 ohm. Under this condition,

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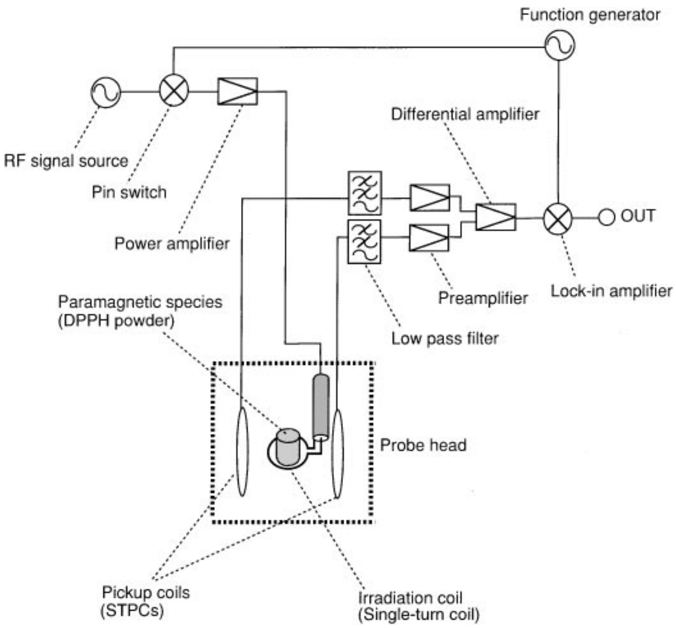

FIG. 1. Block diagram of our LODESR magnetometer. Its probe head consists of a paramagnetic species, an irradiation coil, and a pickup coil.

the single-turn coil can be used over a wide band because the reactance of the inductor is always smaller than 50 ohm at frequencies under 700 MHz (*9*). The RF reflection level in the single-turn coil was investigated by using a network analyzer (Model No. 8714C, Hewlett-Packard, Palo Alto, CA; frequency range, 0.3 to 3000 MHz). The change in the reflection levels between 20 and 300 MHz was less than 0.4 dB. The DPPH powder was inserted into the center of the single-turn coil.

A pair of saddle-type pickup coils (STPCs) (*7–9*) was used to detect the change in magnetization in DPPH. The single-turn coil was inserted into the center of the pair of STPCs.

Measurements of Static Magnetic Fields

The probe head of the LODESR magnetometer was inserted into B_0 to measure it. B_0 was generated using a commercially available electromagnet (modified RE3X, JEOL, Tokyo, Japan). Before this experiment, the magnetic field at the center of the electromagnet was calibrated using a conventional gaussmeter (ML-IV, Magnet Laboratories, Inc., Tokyo, Japan; field range, 1 to 3000 mT; error, ±1.5%).

Figure 2 shows the results of the wide-range measurements for B_0 using the LODESR magnetometer with a single probe head (frequency sweep width, 270 MHz). When the B_0 strengths were set at 2.6 (Fig. 2a), 4.1 (Fig. 2b), 5.6 (Fig. 2c), 7.1 (Fig. 2d), and 8.6 mT (Fig. 2e), the LODESR signals of DPPH appeared at frequencies of 71.9166 (Fig. 2a), 115.9781 (Fig. 2b), 158.5998 (Fig. 2c), 200.7369 (Fig. 2d), and 243.8431 MHz (Fig. 2e), respectively; and the B_0 strengths that were computed from these frequency were 2.5645 (Fig. 2a), 4.1358 (Fig. 2b), 5.6556 (Fig. 2c), 7.1582 (Fig. 2d), and 8.6954 mT (Fig. 2e). These values were in accordance with the set B_0 within the margin of error of the gaussmeter (±1.5%) (*11*). This shows that when the LODESR magnetometer is used without changing the probe heads, it is possible to measure B_0 over a wide range (from 2 to 9 mT).

The signal amplitudes in Fig. 2 are roughly proportional to B_0 with a deviation of ±15%. For longitudinal detection, the signal amplitude is directly proportional to the B_0 strength when a constant magnetic field of radio waves (B_1) is applied (*9*). In a lower frequency band (less than 250 MHz), B_1 is almost constant when a constant power is applied. The degree of deviation from the proportional relationship between the LODESR signal amplitude and B_0 is in accordance with variations in the RF reflection level of the single-turn coil and variations in the gain of the power amplifier that is connected to the single-turn coil (i.e., variation in applied power) at different frequencies. The variation in signal amplitudes can be compensated by calibration (*9*).

There is a small signal at ca. 107 MHz in all of the spectra in Fig. 2. This is not an ESR signal because it appears at the same frequency despite applying different B_0. The current in a single-turn coil (i.e., B_1 strength), the diameter of which is relatively small, approximates to the short current in a transmission line over a wide RF range (*9*). However, the appearance

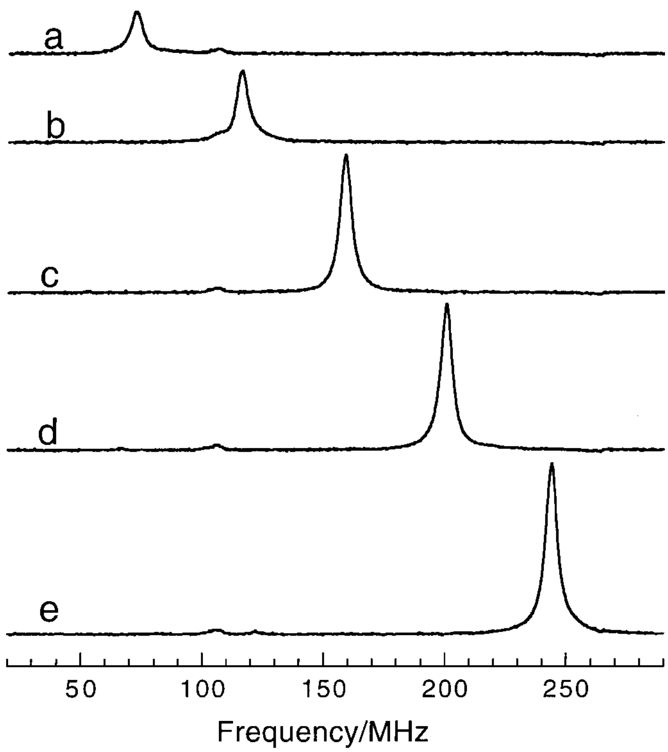

FIG. 2. Experimental results for wide-range measurements of B_0 strength using the LODESR magnetometer with a single probe head. B_0 was set at 2.6 (a), 4.1 (b), 5.6 (c), 7.1 (d), or 8.6 mT (e). The instrument settings for wide-range measurements were start and stop frequency in sweeping, 20 and 290 MHz; sweep time, 30 s; average power, 0.4 W at 290 MHz.

of standing waves is unavoidable because the output impedance of the power amplifier that is connected to the single-turn coil is not perfectly matched to 50 ohm. Therefore, it is presumed that the radio waves with an augmented B_1 at a frequency where standing waves occur are rectified and appear as a small signal in Fig. 2 despite the low pass filter connected between the pickup coils and preamplifier. Such a signal was observed at a different frequency when the length of the transmission line was changed. This supports the explanation given above.

In B_0 (the same as that under the condition shown in Fig. 2d), the narrow-range measurements (frequency sweep width, 4 MHz) were made using the LODESR magnetometer. The LODESR spectrum obtained near its peak for a bandwidth of 1 MHz was fitted by the method of least squares (Fig. 3). The precise B_0 strength was obtained from the resonant frequency, which was derived from the peak point of the fitted curve. Using this measuring method, the standard error, calculated from five independent determinations, was $\pm 0.017\ \mu$T (i.e., $\pm 2.4 \times 10^{-6}$). This value is smaller than that obtained with a commercially available ESR magnetometer (*12*).

The *g*-value of DPPH was measured to an accuracy of ± 50 ppm at 10 mT by calibrating B_0 with an NMR magnetometer (*10*). Thus, the absolute value of B_0 measured with DPPH in this study was to five significant figures. However, reproducibility in measuring B_0 by the LODESR magnetometer mainly depends on the accuracy of the RF signal source because temporal changes in the *g*-value are negligible. The high reproducibility of our magnetometer (error, $\pm 2.4 \times 10^{-6}$) is not inconsistent with the high accuracy of the RF source (frequency error, less than 2×10^{-8}/day) (*13*). In the conventional ESR magnetometer,

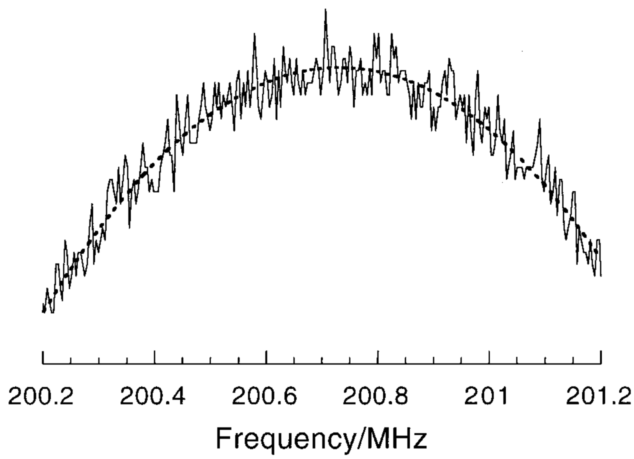

FIG. 3. Example of narrow-range measurements of B_0 (the same as that under the condition shown in Fig. 2d) by using the LODESR magnetometer for a precise determination. The instrument settings were similar to the wide-range measurements (see Fig. 2), except for the sweep width, which was 4 MHz. The LODESR spectrum obtained near its peak for a bandwidth of 1 MHz was fitted by the method of least squares (solid line, raw data; dashed line, fitted curve). The precise B_0 strength was obtained from the resonant frequency, which was derived from the peak point of the fitted curve.

signals are detected with magnetic field modulation (*2, 11*), so the magnetometer itself perturbs B_0. Thus, the reproducibility with the ESR magnetometer would be limited by other conditions (such as the accuracy of the magnetic field modulation) in addition to the accuracy of the RF source.

In this study, a LODESR magnetometer for low fields was newly introduced. This is the first study to apply the LODESR method to measure B_0. In a conventional ESR magnetometer, B_0 is determined from radio wave absorption under ESR irradiation. In applying the LODESR magnetometer, it is determined from the longitudinal change in magnetization under ESR irradiation. Its main advantage over a conventional ESR magnetometer is that a wide range of B_0 can be measured without changing the probe head.

EXPERIMENTAL

RF Irradiation

The radio waves were generated by a synthesized oscillator (Model No. MG3633A, Anritsu, Tokyo, Japan; frequency range, 10 kHz to 2700 MHz; frequency error, less than 2×10^{-8}/day), on/off modulated by a pin switch (ZYSWA, Mini Circuit, New York; frequency range, DC to 5 GHz; switching time, 5 ns) and a function generator (Model No. AFG320, Sony-Tektronix, Tokyo, Japan; frequency range, 0.01 Hz to 16 MHz), amplified by a power amplifier (Model No. A1000-1S-M, R&K, Fuji, Japan; gain, ca. 20 dB; output impedance, 50 ohm; frequency range, 20 to 800 MHz; max power, 1 W), and applied to the irradiation coil. The duty factor in the on/off modulation of the radio wave was 0.5.

Pickup Coils

A pair of STPCs (*7–9*) was used to detect the change in magnetization in DPPH. Each coil of the pair was constructed from 15 turns of copper wire (0.3 mm in diameter). The outer diameter of the coil measured 30 mm. These coils were pasted onto a cylindrical quartz glass tube (38 mm in outer diameter).

LODESR Signal Detection

The longitudinal change in magnetization induced a signal in the pair of STPCs at a modulation frequency of 1.3 MHz. LC matching circuits were used to match the impedance of the pickup coils with the input impedance of the preamplifiers at the modulation frequency. The *Q*-value of the STPCs is 22. To avoid rectifying the modulated RF, a low pass filter (Model No. SLP-5, Mini Circuit; cutoff frequency, 6 MHz) was connected between the pickup coils and the preamplifier. The signals from the right and the left STPCs, which have opposite polarities, were amplified separately by two preamplifiers (Model No. SA-230F5, NF Corp., Yokohama, Japan; gain, 46 dB; input impedance, 50 ohm, frequency range, 1 kHz to 100 MHz; noise figure, 0.6 dB). The difference in outputs from the preamplifiers was amplified by a differential amplifier (Model No. 5305, NF Corp.; frequency range, DC to 10 MHz) to reduce common mode noise. The LODESR signals were detected by a lock-in amplifier (Model No. 5202, PARC, Princeton, NJ; frequency range, 0.1 to 50 MHz) at the on/off modulation frequency.

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REFERENCES

1. F. Hartman, *IEEE Trans. Magn.* **8,** 66 (1972).
2. H. Gebhardt and E. Dormann, *J. Phys. E.* **22,** 321 (1989).
3. D. Duret, M. Beranger, and M. Moussavi, *IEEE Trans. Magn.* **28,** 2187 (1992).
4. J. K. Hulbert, *J. Phys. E.* **9,** 283 (1976).
5. A. Colligiani, D. Leporini, M. Lucchesi, and M. Martnelli, *in* "Electron Magnetic Resonance of Disorder Systems" (N. D. Yordanov, Ed.), pp.16–37, World Scientific, Singapore, 1991.
6. I. Nicholson, F. J. L. Robb, and D. J. Lurie, *J. Magn. Reson. B* **104,** 284 (1994).
7. H. Yokoyama, T. Sato, N. Tsuchihashi, T. Ogata, H. Ohya-Nishiguchi, and H. Kamada, *Magn. Reson. Imaging* **15,** 701 (1997).
8. H. Yokoyama, T. Sato, T. Ogata, H. Ohya-Nishiguchi, and H. Kamada, *J. Magn. Reson. B* **129,** 201 (1997).
9. T. Sato, H. Yokoyama, T. Ogata, H. Ohya-Nishiguchi, and H. Kamada, *Appl. Magn. Reson.* **16,** 33 (1999).
10. T. Shirai and M. Kosukegawa, *JEMIC Tech. Rep.* **29,** 47 (1994).
11. See the catalog of ML-IV, Magnet Laboratories, Inc., 3-43-3 Yamato, Nakano, Tokyo, Japan.
12. See, for example, the catalog of EFM-30AX, Echo Electronics, 4-8-41 Kashiwa, Shiki, Japan.
13. See the catalog of MG3633A, Anritsu, 5-10-27 Minamiazabu, Minato, Japan.